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PROGRESS OF ANALYTICAL CHEMISTRY IN THE USSR FOR 1949

Yu. A. Klyachko

Development of the Theory

Quite a number of works on the theory of chemical analysis were published in 1949. I. V. Tananayev and his coworkers continued their works on physicochemical analysis of systems having significance in analytical chemistry. They investigated systems of K_2PdCl_4-KI-H_2O (2), CoSO-$LiFeCy_6$ - Na_4FeCy_6, Cs_4FeCy_6 - H_2O (3), the reaction of forming copper rubeanate (4), the co-agulation kinetics of the PdI_2 precipitate (5). The second of these works gives an interesting generalization that cobalt salts form simple ferrocyanides of cobalt in their reactions with ferrocyanides of alkaline metals, ions of which have small relative volume and are well hydrated (Li, Na); double salts are formed in reactions with ferrocyanides of other alkaline metals. Thus, I. V. Tananayev's works lead to a series of generalizations which have great significance not only for developing individual analytical methods but also for clarification of general conditions for realizing analytical reactions. I. V. Tananayev and S. Ya. Levitman (4) established the composition of copper rubeanate and suggested, on the basis of their investigations, the photo-turbidimetric method for the determination of copper the example of developing individual analytical methods with the aid of physicochemical analysis of reactions.

In his works on palladium compounds I. V. Tananayev evaluates the significance of specific colloidal phenomena in the process of precipitate formation. Ignoring these phenomena has been a weak point in I. V. Tananayev's works as it was indicated in the previous review (1). Under estimation of adsorption phenomena and interpretation of all quantitative

relations only from the viewpoint of structural stoichiometric relationships may lead to erroneous conclusions. The possibility of such mistakes may be illustrated by the work of Sh. T. Talipov and I. L. Teodorovich (6) who found, in the potentiometric determination of NaF by titration with $FeCl_3$, that this system forms the compound $2\ FeF_3 \cdot 6NaF$ with certain excess of iron in the equivalence point, whereas I. V. Tananayev and M. E. Daychman attribute the formula $2FeF_3 \cdot 5NaF$ to the same compound. Probably, the latter authors considered the adsorption interaction as chemical stroichiometric one. Especially underestimation of adsorption-colloidal interaction is dangerous in employing the light absorption process as a method of physicochemical analysis. Thus, in the last of the above mentioned works I. V. Tananayev considers one of most essential colloidal processes - the coagulation process. Data obtained on the system composition, which gives the maximum light scattering effect have a great principle significance because they lead to constructing the theory of turbidimetric analytical methods.

The other large series of works continued in 1949 is studying the influence of complex formation on oxidizing-reducing potentials of systems by V. S. Syrokomskiy and coworkers (7-10). These articles gives a great deal of new experimental material which may be used in the interpretation of the chemical mechanism of many reactions and in generalizing the laws of elementary processes of the chemical form of motion.

There are three theoretical works which may be considered as most important next to the two series described above: the work of Ya. P. Gokhshteyn (11) on the theory of polarographic analysis, the work of N. A. Izmaylov (12) on the application of anhydrous solvents in analytical chemistry and the work by V. M. Chulanovskiy, A. F. Adrianov and B. I. Rubinovich (13) dedicated on the colorimetric analysis method of poly component mixtures.

Several theoretical works have to be mentioned as having certain importance. Two works by S. D. Shargorodskiy and Ya. A. Fialkov (14) and by S. D. Shargorodskiy (15) investigated thermal decomposition of metal sulfates of the second group of the periodic system. The first article is dedicated to sulfates of beryllium, magnesium, calcium, strontium and barium and the second one - to sulfates of zinc and cadmium. The therographic method was employed for establishing the temperatures for eliminating water of crystallization and for polymorphous transformations.

I. A. Korshunov and M. K. Shchennikov (16) began an essential work on detailed characteristics and study of various conditions for polarographic reduction of inorganic ions.

A. T. Pilipenko (17) established a series of solubility products for xanthogenates of heavy metals.

V. F. Toropova and F. M. Batyrshina (18) determined compositions of acetate complexes of lead using the polarographic method.

New Methods

The first group of new methods consists of new methods of chemical analysis which are already known but yet are not sufficiently developed. Among these methods the greatest attention was paid to the chromatographic adsorption analysis and polarography with amperometric titration.

Several works in the field of chromatography were published. The general review of chromatographic analysis is given by M. M. Senyavin (19). Distribution and ion-exchange chromatography is excluded from this review, because they were discussed in other works (see the author's review of works for 1948). The review article by M. A. Fuks (20) is dedicated to the progress of the chromatographic method in organic chemistry. Systematic investigations of Y. Y. Lure and M. A. Filippova on application of organolites in analytical chemistry were continued. The published article (21) deals with methods for determination of sulfur, phosphorus and arsenicin nickel and copper. T. B. Capon and Ye. M. Capon (22) acheived

the solution of the difficult problem - separation of cobalt and nickel - by obtaining a special kind of permutite.

Interesting works on studying the adsorption method of analysis are conducted in the Ukraine. M. Ya. Romanova and I. P. Girko (23) staged works on determination of the organolite capacity what has a basic significance for technological application of organolites but also is essential in analytical chemistry.

B. G. Savinov and L. F. Grinberg (24) studied the problem of selecting and activating the adsorbents for chromatographic separation of carotene and products of its oxidation. They found, for example, that the best adsorbent for separating the natural isomers of carotene is MgO obtained f from $MgCO_3$ by heating at 600-700 degrees for 4 hours. For products of carotene oxidation, the best results are received by application of CaO obtained after thermal treatment.

A series of works were dedicated to polarography and amperometric titration. Fundamental works on polarography have a purpose to improve the procedure of this method by elimination of maxima on polarographic curves.

Several works on this subject were written by Ye. M. Skobets with collaborators (25-28). E. M. Skobets and P. P. Turov (25) employed the solid electrode and electrochemical depolarization. Ye. M. Skobets and N. N. Atamanenko (27) investigated particular advantages of the solid electrode, the platinum rotating microelectrode, used as an anode since mercury has a very low potential of anodic dissolving. Ye. M. Skobets, P. P. Turov and V. D. Ryabokon (26) achieved the elimination of polarographic maxima by heating and Ye. M. Skobets and N. S. Kavetskiy (28) attained the same result employing the mercury dropping electrode with forced release of a drop. The work of V. A. Tsimmergakl (29) contributes to the same method.

Yu. S. Lyalikov and R. I. Glazer continued their work on application of the solid dipping electrode in polarography (30).

The Gor'kiy school of polarographers continued their investigations on application of polarography in organic chemistry (31-33). One of these works describes application of the multiple-stream mercury dropping electrode of unique shape, but no information is given in regard to its features and advantages.

The amperometric titration is a promising part of polarography, and an activity in this field is represented by the large work of O. A. Songina, A. P. Voyloshnikova and M. T. Kozlovskiy (34) which is the first communication of the new series of works. This paper reveals advantages of amperometric titration in respect to potentiometry, conductometry and polarography; it proves the possibility of determining small quantities of elements and the low concentrations of ions in the presence of high concentrations of other ions; it also demonstrates the simplicity of equipment and independence from the capillary characteristic, concentration and temperature. There are several unique moments in this work: titration with the "universal" reagent - potassium iodide which may be used for the precipitation reactions as well as for oxidizing-reducing reactions and for complex formation; application of the mercury iodide comparison electrode. Some practical conclusions are also made: the possibility was established for determining small quantities of silver in the presence of lead, tellurium, mercury and bismuth without superposition of the external electromotive force.

The article by V. I. Kuznetsov (35) deals with application of organic reagents in analysis. It generalizes materials on coloration reactions, named by the author as "solid phase" reactions, in which precipitated compounds have a color different from the solution color of the same substances. The author demonstrates that such reactions especially often occur at interaction of numerous organic substances with inorganic ions. Also, the chemical interpretation is given for the mechanism of some reactions which occur on application of adsorption indicators.

Yu. A. Chernikhov and B. M. Dobkina conducted an investigation of possibilities for analytical application of sodium diethyl dithiocarbamate (36). They described, for the first time, the carbamates of gallium, tellurium and rhenium, suggested conditions for analytical application of diethyl dithiocarbamate and established the analogy between carbamates and sulfides.

The work by B. N. Afanas'ev discusses utilization of chloramine as a quantitative oxidizer for determination of organic compounds - aldehydes, ketones, glucose and others (37).

The new method for determination of molybdenum in steels with the aid of beta-naphthoquinoline is suggested by R. B. Golubtsova and F. M. Shemyakin (38).

The group of methods which are subject to further development also includes the methods of organic microanalysis, to which M. O. Korshun and V. A. Klimova contributed their article (39) on rapid combustion of nitro-compounds without application of lead dioxide, and several works on carbide analysis (40-42). The intensive investigation in this latter field is continued. The group of entirely new methods embraces the following works: determination of copper in small quantities with the aid of hydrazine sulfate in volumetric and gravimetric variations, by Tt. G. Raykhinshteyn (43); determination of the concentration of aqueous salt solutions by titration with alcohol based on the classic investigations of I. M. Secnenov on dependence of mutual solubility of water and nonelectrolyte on salt concentration in solution, by S. I. Spiridonova (44); separate semi-microdetermination of hydrocarbon gases of the methane series, by M. G. Gurevich (45).

Qualitative Analysis

Kh. N. Pachinok and V. Ya. Pochinok suggested a new sensitive reagent for magnesium (46); alkali or alkali earth metals and lead do not interfere with application of this reagent. The reagent 3-phenyl-1-paramitrophenyl-3-oxytriazene synthetized by the authors may also be use in colorimetry.

L. M. Kulberg and R. B. Liokumovich (47) suggested the color reaction for a barium ion, B. N. Afanas'ev and A. V. Ural'skaya (48) developed the color reaction with chloramine for bismuth.

E. A. Bitovt (49) studied the conditions for obtaining and the structure of the compound formed at reaction of zinc ions with pyramido-rhodanide reagent. S. I. Gusev developed the method for detecting cadmium in the presence of copper with an antipyrine bromide reagent (50).

A. I. Busev (51) solved the problem of detecting osmium in the presence of ruthenium with the aid of thicurea and diethyl dithiophosphoric acid.

B. S. Tsyvina published two works (52, 53) in which she investigated microchemical determinations of sodium and calcium with the aid of nickel-uranyl-acetate and nickel-cobalt-nitrite complexes using them for colorimetric determination.

Application of New Methods

Numerous works were published in 1949 describing utilization of new methods of analysis in various fields of industry, agriculture, medicine and others. The experience of employing the amperometric, photoelectric and dropping methods received a specially wide attention . The amperometric titration found its application in analysis of electroplating baths. This application is described in two works. In the first one V. F. Toropova, Ye. A. Zim'kin and A. A. Pospelov (54) determine amperometrically the sulfates in the presence of a chloride-ion using lead nitrate for titration. Authors of the second work, N. C. Chovnyk, N. N. Kuz'mina, A. N. Galkina and B. Ya. Starik (55) conducted more complete analysis determining admixtures by polarographic methods and basic components - with the aid of amperometric titration with the dropping mercury electrode (Ni, Cu, Zn, SO_4^{-2}),

K. A. Vasil'ev and S. Ya. Getsova developed the method for amperometric determination of sulfate-ion in the presence of large quantities of fluoride in cryolite, aluminate solutions, iron fluoride and so forth (56).

G. S. Laxina suggested the method for the amperometric determination of small amounts of fluoride . the reaction of forming thorium fluoride (57).

T. T. Khametov (58) studied amperometric determination of iron, chromium and vanadium in ores, cast iron and steels. He investigated the effect of temperature on the position of the equivalence point. The gel of silicic acid was used as a key compound for high temperatures.

Photoelectric methods are in use for a long time but the works on this subject published in 1949 demonstrate the possibilities of standardizing application of these methods and show directions for further developing the field of their use.

A great deal of attention to application of photoelectric methods was paid in the pamphlet by T. N. Kazlyayeva, M. A. Petrova and M. N. Sokolova (59), described methods for determining harmful vapors and gases in the air of industrial establishments. These methods were developed in the Leningrad Institute of Labor Protection (LIOT). Authors discuss the methods of determining chlorine, ozone, nitrogen dioxide, sulfur dioxide, arsine and other arsenic compounds, fluorine, benzene, alcohols of the aliphatic series. Using the photonephelometric method, they determined hydrocyanic acid, sulfur dioxide and sulfuric acid mist in their simultaneous presence and also lead, mercury, turpentine, dichlorethane, trichlorethylene, tetraethyl lead.

D. P. Shcherbov (60) developed in detail the method for photocolorimetric determination of cobalt in iron-nickel ore with application of the nitroso-R-salt.

O. S. Feldman (61) investigated the methods of photocolorimetric determination of albumin, hydrocarbons in foodstuffs, chloropicrin in grain and so forth.

The work by R. Ye. Osherovich and G. V. Rabovskiy (62) on the phototurbidimetric determination of SO_2 in the presence of nitrogen oxides indicates the possibility of using this method for analysis of nitrose gases, but this was not verified in practice.

The drop method without chips in application to sorting ferrous and nonferrous alloys is described in the special manual by Ye. I. Nikitina (63) who has been working on the method and its employment in industry since 1939.

A. G. Kon'kov (64) applied the method without chips to determination of silicon, manganese, chromium, nickel, vanadium and molybdenum in steel.

Problems of using the drop analysis method in the rubber industry are discussed in works by L. M. Kulberg, G. A. Blokh and Ye. A. Colubkova (65) and by S. I. Burmistrov (66). Both works supplement each other and partially use the results of previous works on the subject.

Introduction of the chromatographic method of analysis in the practice of the metallurgical plant laboratory was the subject of two articles by Yu. I. Vsatenko and O. V. Datsenko (67, 68). They suggested the method for determination of phosphorus in ferrophosphorus and copper phosphide and also the method for determination of molybdenum in ferromolybdenum with the aid of sulfocarbon on which molybdenum is absorbed and then washed out with alkali.

Several serious works clarify the application of problems of the potentiometric method. B. G. Ivanov and S. M. Bezyayko (69) offered the potentiometric method for determination of aluminum in bronzes and steels and developed the laboratory technique convenient in mass analysis.

A. I. Busev and N. I. Dmitrieva (70) developed the noncompensating method for determination of manganese in nonferrous alloys. N. A. Glebov (71) confirms the practical expediency of this method.

A good example of introducing new methods into analysis practice is given by the work of Academician G. S. Landsberg and Academician B. A. Kazanskiy with their coworkers on utilization of the complex - chemical and optical method for detail investigating of the individual composition of gasolines (72). They employed the fractionation, group analysis and investigation of spectra of combination light scattering of obtained fractions. Any component may be determined at content in the fraction not less then 1 - 3 percent.

History of Analytical Chemistry in Russia and USSR

The first article on history of analytical chemistry in Russia was written by K. B. Yavorskiy (75). A. Kh. Batalin (74) outlines the history of microchemical analysis in Russia.

The article by V. A. Tsimmergakl (76) establishes the priority of Russian researcher V. A. Tyurin in discovering the "Nernst formula" for concentrating amalgam chains.

G. S. Vozdvizhenskiy dedicates his article (77) to Professor of the Kazan' University F. M. Flavitskiy in which he states that F. M. Flavitskiy was a pioneer of investigating the reactions in solid phases and of that field of microchemical analysis which later was developed into drop analysis.

Conclusion

Development of analytical chemistry in the USSR is progressing. But there is a very little discussion or criticism of methods, investigation trend, articles and books. Only one article written by Y. Y. Lur'e and A. V. Yevlanova (34) may be related to this category of works. Authors investigated the method for determination of pH with organic solvents suggested by Yu. V. Karyakin, criticized his formulas. But such cases are very few. The author of present review expresses his opinion that proper progress in analytical work and literature is impossible without further development of criticism and self-criticism.

Organization and development of numerous analytical schools and trends should be especially noted in entire area of the USSR. Schools and serious analytical groups of investigators are formed in Kiyev, Sverdlovsk, Khar'kov, Alma-Ata, Vladivostok, Kazan', Molotov, Gor'kiy, Ivanov, Saratov, Chernovitsy and others

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